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Correlation between microstructure and microhardness in a friction stir welded 2024 aluminium alloy

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Abstract

A 2024-T351 aluminium alloy has been friction stir welded and the microstructures investigated. An inner HAZ hardness minimum was a result of an overaged S phase, whereas an outer minimum was believed to be due to precipitate dissolution. An interjacent HAZ maximum was attributable to the presence of very fine S phase precipitates. The nugget zone contained ~4 μm grains and complex dislocation structures.

Keywords: Friction stir welding; Aluminium alloys; Precipitation; Hardness; TEM

1. Introduction

Friction stir welding has been well documented in recent times as an economically viable solution to the problem of joining hitherto difficult to weld aluminium alloys (e.g. [1]). In particular it has attracted the attention of the aerospace industry, due to potential weight and cost savings, combined with favorable retention of properties (e.g. [2]).

The details of the process have been described elsewhere (e.g. [3]), and in essence consist of a rotating pin (connected to a shoulder) which is forced into and then translated along the join line. This results in plastic deformation in the vicinity of the pin and significant levels of frictional heating, due to contact between the tool pieces and material. Exceptionally high strain levels (i.e. >40 [4]), experienced in what is termed the 'nugget zone', have been found to result in very fine grain sizes (i.e. <10 μm [5]). Previous work relating microstructure and properties, in a precipitation hardenable 6063 aluminium alloy, has been undertaken by Sato et al. [6] and also by Ortelt et al. [7]—for a 2195 aluminium alloy. The latter authors found differences in the dislocation density within the nugget zone grains (a dynamically recrystallized zone), together with incipient formation of sub-grain boundaries. Interestingly, they also observed a reduction in hardness within the HAZ, at ~15 mm from the centre of the weld nugget.

An understanding of the microstructural 'mechanisms' occurring during and after the welding process is important if resultant weld microstructures and associated mechanical properties are to be optimized. The aim of the present work—which is part of a larger study looking at modeling of the material flow during the process [8]—is to investigate and correlate the various weld microstructures and micro-hardness using transmission

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electron microscopy (TEM), scanning electron microscopy (SEM) and electron back-scattered diffraction (EBSD) techniques.

2. Material and experimental methods

The base material used in this study was a 2024-T351 aluminum alloy in the form of 15 mm plate (grain size ~56 μm). Vickers micro-hardness measurements of the base material were found to be ~135H_v, regardless of location. Samples of this plate were tooled down to 10 mm thickness and friction stir welded by EADS CCR (France). The joint was sectioned in the transverse plane, enabling microstructures produced within specific areas of the welded material to be examined. Specimens for TEM studies were prepared by spark eroding discs of 3 mm diameter from the nugget and HAZ regions of the weld. Thin foils were then produced using a twin-jet polisher containing a 30% nitric acid and 70% methanol solution at −30 °C and 12 V, and specimens were then examined using a Phillips CM200 operating at 200 kV. SEM and EBSD specimens were mechanically polished using standard techniques, prior to electropolishing under the same conditions as that of thin-foil preparation. Specimens were then examined in a JEOL JSM-6500F FEG-SEM operating at 20 kV. Orientation maps were obtained from electropolished SEM specimens using the HKL Channel EBSD acquisition system. In order to investigate the variation in mechanical properties, Vickers micro hardness (H_v) measurements were made across the transverse plane using a Matsuzausa MXT 70 micro-hardness tester (100 gf load) at mid thickness.

3. Results and discussion

Fig. 1 shows part of the hardness profile across the transverse plane of the weld on the retreating side (together with the locations used for microscopic examination). Moving out from the edge of the nugget zone (the TMAZ extended out to ~8 mm), the HAZ can be seen to contain an inner and outer minimum, together with an interjacent maximum-located ~12, 27 and 18 mm from the join line respectively. The hardness in the nugget zone can be seen to fluctuate in the range 120–130H_v, which may be due to experimental error when considering the similar variation seen at distances >35 mm from the join line (i.e. approaching the base material (135H_v) towards the edge of the HAZ). Although not investigated, it should be noted that this variation in hardness may be due to the 'onion ring' structure of the nugget zone and associated precipitate distribution (e.g. [9,10]).

Both the central and retreating sides of the nugget zone (locations 1 and 2 in Fig. 1) were found to contain

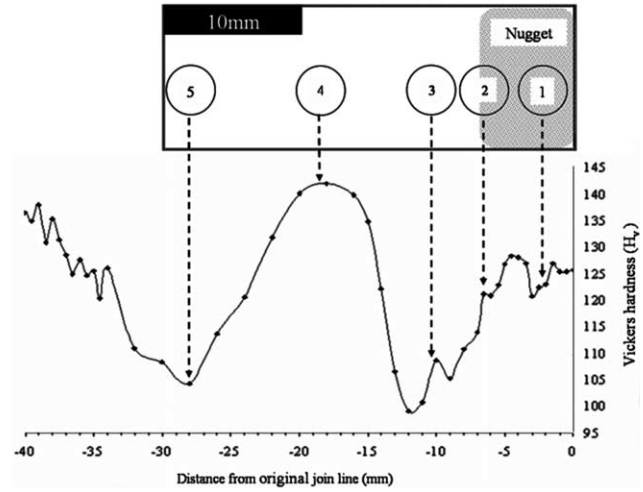

Fig. 1. Schematic of weld transverse plane (retreating side) showing locations used for TEM examination together with their associated hardness values.

a very fine equiaxed grain size (~4 μm), as shown by the bright field TEM image in Fig. 2. EBSD orientation maps gave a slightly higher grain size of ~5 μm (due to low angle grain boundaries (LAGBs) below 2° being omitted) as shown in Fig. 3. This microstructure is typical of that found in the nugget zone (e.g. see [5]), and as generally reported in previous investigations, the presence of a high proportion of high angle grain boundaries (HAGBs) in the nugget zone (e.g. Fig. 3) contributes to hardening through the well known Hall–Petch relationship. A number of these fine grains were found to contain a high dislocation density, whereas others appeared to have rather low densities. In some instances a number of helical dislocation structures were observed within

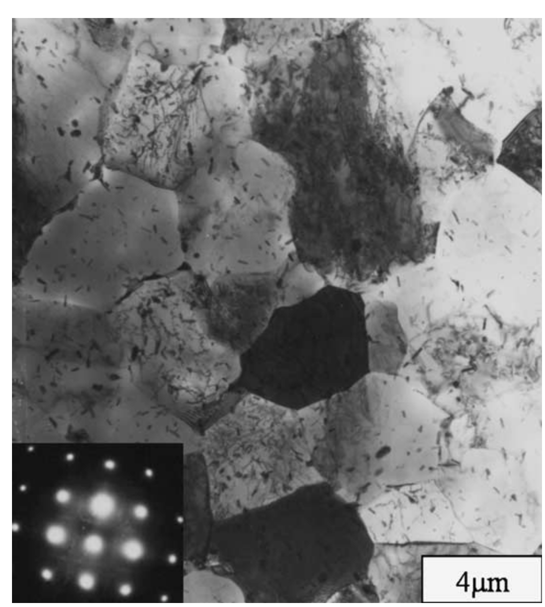

Fig. 2. TEM micrograph of central nugget zone (location 1 in Fig. 1).

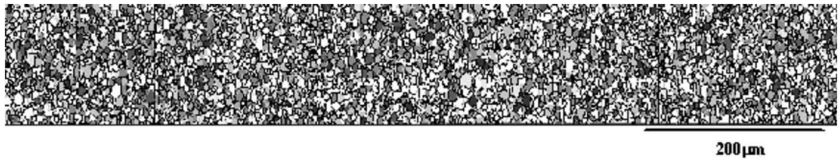

Fig. 3. EBSD orientation map of nugget zone: LAGBs (2–15°) shown as grey lines, HAGBs (>15°) as black lines and grains with the same orientation shown with same grey level.

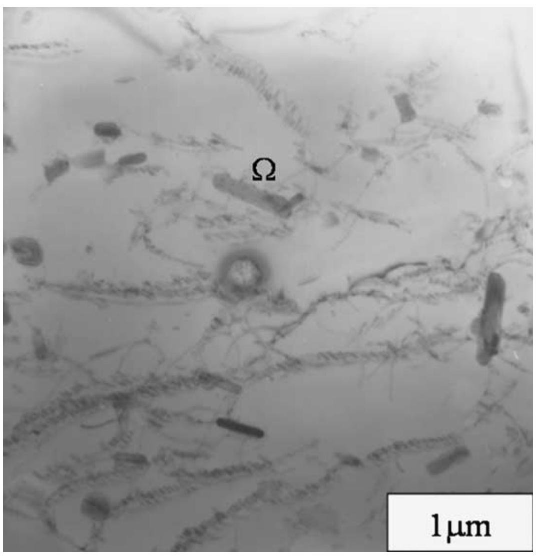

Fig. 4. TEM micrograph showing interior detail of a central nugget zone grain (location 1 in Fig. 1).

the grains, as shown in Fig. 4. These structures were typically ∼1–2 μm in length and frequently aligned along low index crystallographic directions. In a previous investigation on a friction stir welded 7050-T651 alloy, helical dislocation structures were seen in the nugget zone and were believed to have formed during the 'thermal quench' from the weld to ambient temperature [11]. Selected area diffraction patterns (SADP), with the beam parallel to $[001]_{Al}$ (inset Fig. 2), showed diffuse streaks through 210 reflections along [001] directions. This was seen to be indicative of the presence of very fine scale S (Al_2CuMg) precipitates (no distinction is made between the S phase or its precursor variants in this study). Evidence of larger Ω (Al_2Cu) type precipitates (indicated in Fig. 4) was also seen from SADPs, with particle reflections seen at {111} positions. The morphologies of these Ω type precipitates were seen to be similar to those observed in the HAZ of a variable polarity plasma arc (VPPA) welded 2024-T351 aluminum alloy [12], where they were attributed to a slow cooling effect (see also [13]). It should be noted that the crystallographic relationship between the Ω type precipitates and the surrounding matrix, indicates that they formed during cooling of the material to ambient temperature (the weld was air cooled in the present case) and that the nugget zone has been calculated to reach temperature of ∼460 °C [8] during the weld thermal cycle (i.e. solutionizing and subsequent re-precipitation has occurred.)

EBSD orientation maps of the nugget zone/TMAZ boundary regions for both the advancing (for comparison) and retreating sides of the weld are shown in Fig. 5. The advancing side is characterized by a typically distinct boundary between the nugget and the TMAZ. This contrasts with the retreating side of the weld, where the boundary between the nugget zone and the TMAZ is rather unclear. This is due to the torsion (due to the rotating motion of the tool) and the circumventing (due to the translation motion of the tool) velocity fields having opposite directions on the advancing side, whereas these velocities have the same direction on the retreating side. The HAGBs on the retreating side also appear to have fragmented to a greater extent than those on the advancing side.

The rather different microstructure shown in Fig. 6, corresponds to the inner minimum in the HAZ (position 3 in Fig. 1), where a drop in hardness was seen at a distance of ∼12 mm from the join line (retreating side). Interestingly, a drop in hardness within the HAZ, has also been observed at a comparable distance of ∼15 mm from the centre of the nugget zone in a 2195 aluminium alloy [7]. In the present investigation, the HAZ grain size is considerably larger than that of the nugget zone, and a high density of lath-like precipitates (∼100 nm in length) can be seen within each grain (inset Fig. 6). SADPs revealed the presence of particle reflections at S positions, indicating that coarsening and overaging of the S phase occurred due to the thermal cycle. This is consistent with the drop in hardness seen at this location. Equiaxed dispersoid type particles are visible in Fig. 6, which were confirmed by EDX measurements to be Al-Mn based. Dispersoids (present in the base material) are stable to high temperatures and the thermal cycle experienced within the HAZ would be sufficient to cause some coarsening but not solutionizing. Closer inspection of Fig. 6, revealed the presence of a Precipitate free zone (PFZ) along a grain boundary (i.e. indicative of an overaged microstructure), together with a number of grain boundary precipitates—which have also been observed in a 2195 aluminium alloy [7]. The PFZs were seen to not be a general feature of the microstructure in this part of the weld; although they have been seen in the HAZ of a friction stir welded 7050-T651 aluminum alloy [11].

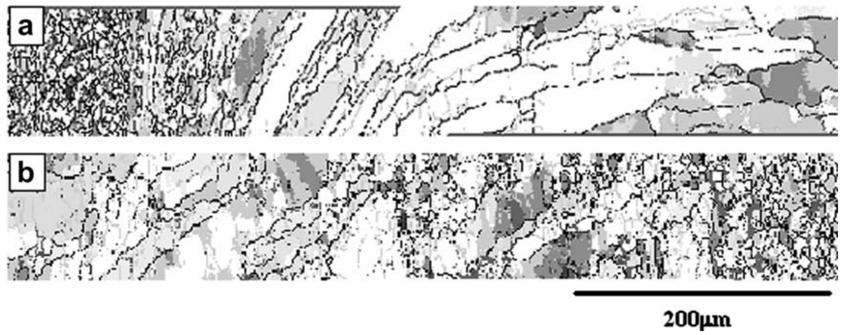

Fig. 5. EBSD orientation maps of the nugget zone/TMAZ boundary regions for both the advancing (a) and retreating sides (b) of the weld.

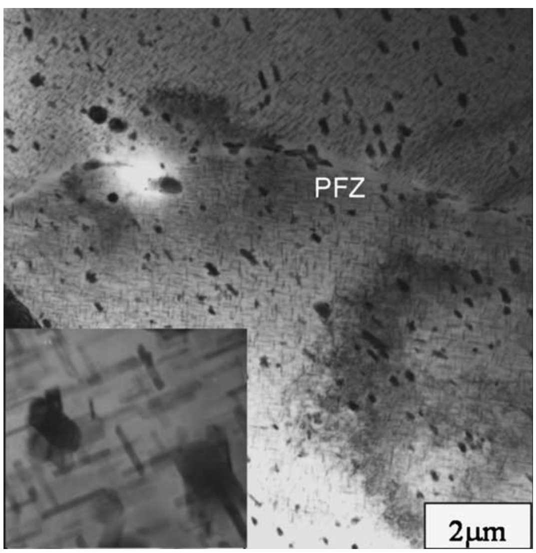

Fig. 6. TEM micrograph from region where inner hardness minimum was seen in the HAZ (location 3 in Fig. 1).

At a distance of ∼18 mm from the join line in the HAZ (location 4 in Fig. 1), the hardness maximum was seen to be due to the presence of very fine S phase precipitates. This is shown by the SADP in Fig. 7 (inset), where diffuse streaks through {2 1 0} reflections along [0 0 1] directions are visible (i.e. the weld thermal cycle has resulted in optimum conditions for aging of the S phase). Relatively large high aspect ratio particles, together with equiaxed dispersoid type particles are also visible. The latter are thought to be base material constituent particles which were largely unaffected by the weld thermal cycle. Interestingly, the base material microstructure—and associated SADPs—were found to be very similar to that shown in Fig. 7.

TEM and FEG-SEM examination of the outer hardness minimum, ∼27 mm from the join line (location 5 in Fig. 1), did not reveal any evidence of fine scale precipitation. The microstructure at this location was found to be very similar to that shown in Fig. 7 for location 4 (when imaged in BF TEM mode). However, inspection of the SADPs did not reveal any particle reflections

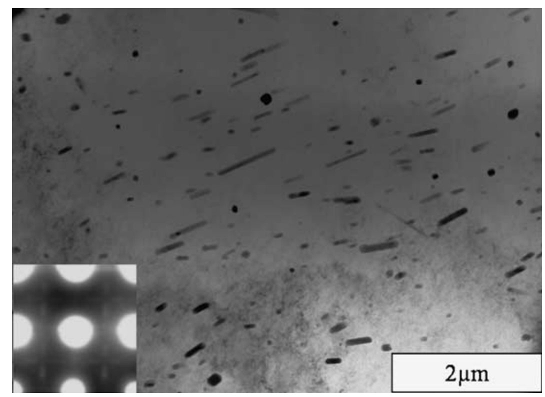

Fig. 7. TEM micrograph from region where hardness maximum was seen in the HAZ (location 4 in Fig. 1).

at S positions, indicating the absence of any fine scale precipitation at this location. A possible explanation may be that room temperature aging occurring during the storage of 2024 type aluminium alloys, through very fine S phase (i.e. GP zone) precipitation, may be significantly slowed down due to a lack of quenched-in vacancies. Then, during the subsequent weld thermal cycle, a comparatively small increase in temperature (estimated to be ∼200 °C [8]) might result in zone dissolution and the subsequent drop in hardness seen at this location.

4. Conclusions

The HAZ on the retreating side of a friction stir welded 2024-T351 aluminium alloy has been found to contain two distinct hardness minima on either side of a maximum. The inner hardness minimum close to the TMAZ was found to be due to coarsening and overaging of the S phase occurring during the thermal cycle. The outer hardness minimum was thought to be due to dissolution of the very fine S phase occurring towards the outer edge of the HAZ. The hardness maximum interjacent to these two minima was seen to be due to the presence of very fine S phase precipitates, and is likely to be a result of optimum aging conditions being achieved. The nugget zone was found to be typically fine grained (~4 μm) and contained complex dislocation structures within the grains, together with evidence that fine scale S and larger Ω phase precipitation had occurred from a solutionized state.

Acknowledgments

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